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A FIELD METHOD FOR THE DETERMINATION OF LEAD IN GLASS USED FOR SHIELDING TELEVISION RECEIVER COMPONENTS

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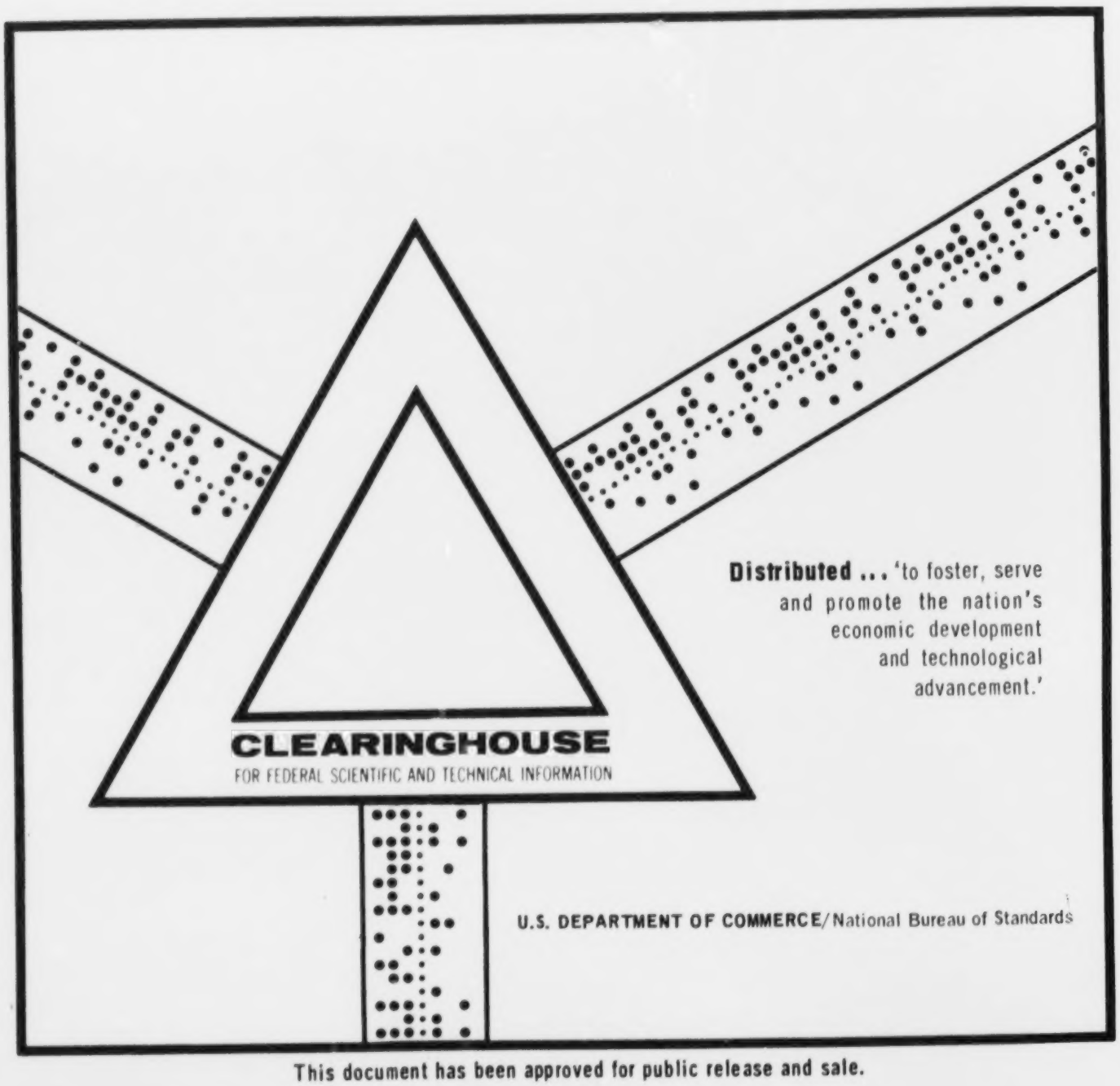
Bureau of Radiological Health
Rockville, Maryland

December 1969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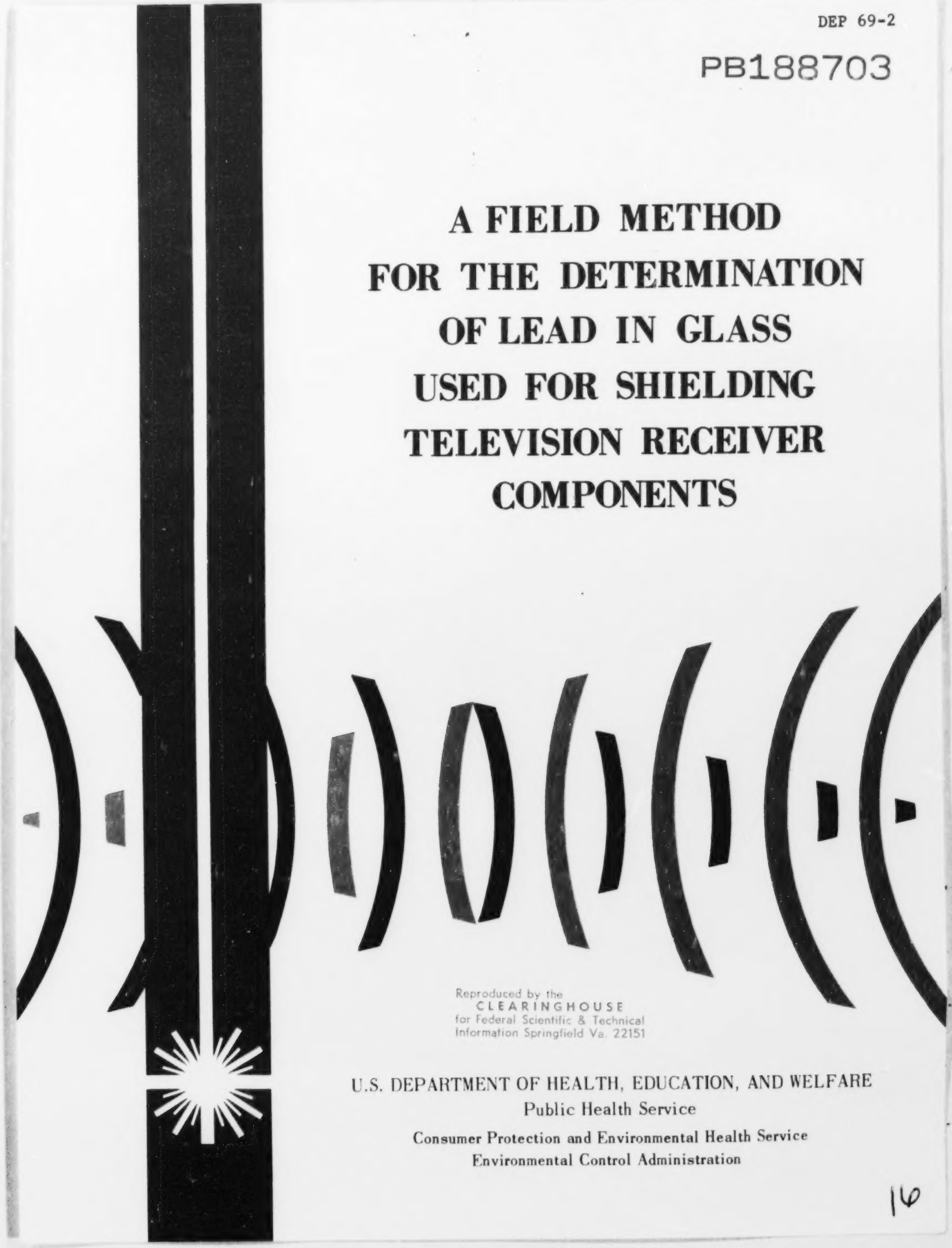

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DIVISION OF ELECTRONIC PRODUCTS

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PB-188 703

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FOREWORD

The Bureau of Radiological Health, formerly the National Center for Radiological Health, continues to carry out a national program designed to reduce the exposure of man to hazardous ionizing and non-ionizing radiation.

Within the Bureau, the Division of Electronic Products (1) develops and administers performance standards for radiation emissions from electronic products, (2) studies and evaluates emissions of and conditions of exposure to electronic product radiation and intense magnetic fields, (3) conducts or supports research, training, development and inspections to control and minimize such hazards, and (4) tests and evaluates the effectiveness of procedures and techniques for minimizing such exposures.

The Bureau publishes its findings in Radiological Health Data and Reports (a monthly publication), the Environmental Health Series, Public Health Service numbered reports, appropriate scientific journals, and Division technical reports.

The technical reports of the Division of Electronic Products (DEP) allow comprehensive and rapid publishing of the results of intramural and contractor projects. The reports are distributed to State and local radiological health program personnel, Bureau technical staff, Bureau advisory committee members, university radiation safety officers, libraries and information services, industry, hospitals, laboratories, schools, the press, and other interested individuals. These reports are also included in the collections of the Library of Congress and the Clearinghouse for Federal Scientific and Technical Information.

I encourage the readers of these reports to inform the Bureau of any omissions or errors. Your additional comments or requests for further information are also solicited.

John C. Villforth
Bureau Director

PREFACE

The "Radiation Control for Health and Safety Act of 1968" (Public Law 90-602) effective October 18, 1968, provides that the Secretary of Health, Education, and Welfare shall establish an "electronic product radiation control program which shall include the development and administration of performance standards to control the emission of electronic product radiation."

In order to control the emission of X-radiation in electronic products, proper shielding in the construction of tubes in color television sets is necessary and the amount of that shielding is an important factor.

This report introduces a methodology for a rapid field test for determining the lead content in glass used for shielding television receiver components as well as glass covering diagnostic X-ray fluoroscopic screens. Information can thus be gathered to determine with relative certainty the ability of the glass to absorb X-rays. Measurements made to show the relative comparison between lead content of glass in envelope and resultant exposure rate are described and results reported.

Robert L. Elder

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Acting Director
Division of Electronic Products

A FIELD METHOD FOR THE DETERMINATION OF LEAD IN GLASS USED FOR SHIELDING TELEVISION RECEIVER COMPONENTS

Harry Levine and Paul S. Ruggera[1]

The transmission of X-rays through the glass envelopes of certain types of television tubes, or through the glass covering X-ray fluoroscopic screens, is influenced by the amount and type of high atomic number elements in the glass. Interest in this aspect of the subject was stimulated by recent findings (1) that color television receivers have been shown to emit X-radiation in excess of NCRP recommendations (2), particularly under certain conditions of operation and construction. These emissions have been found to originate from picture tubes, shunt regulator tubes, and high voltage rectifier tubes (3).

When conducting inspections of equipment where transmission of X-radiation is in question, it is useful for the inspector to know the composition of the glass so that its ability to absorb X-rays may be estimated. Of particular interest would be such items as the shunt regulator rectifier and direct-viewed cathode ray picture tubes used in color television receivers, and glass covering diagnostic X-ray fluoroscopic screens. One can detect substitution of non-leaded glass inappropriate for shielding by means of this test, rather than to perform an X-ray transmission measurement. Additionally, it is sometimes desirable to check the manufacturer's statement on the composition (particularly the lead content) of glass components as these have been found, on occasion, to have been in error.

Lead is the element most commonly used in glass for its radiation attenuation, although barium is often used in the television picture tube faceplate because it causes less coloration and loss of light transmission. A rapid, semi-quantitative method for the detection and estimation of lead in glass, suitable for use in the field, has been developed. It is based on the action of a mixture of hydrofluoric acid and sodium iodide on lead contained in glass and ceramics (4). Important features of this test are that the reaction can be evaluated in a matter of minutes in the field without causing visible damage to the specimen, and the elimination of the conventional chemical analysis that is generally performed in the laboratory. It will show at once whether lead is present in appreciable amounts or is essentially absent.

Dilute hydrofluoric acid is used to dissolve a small section of the glass to be tested and is left in contact with the glass surface for a

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[1]Mr. Levine and Mr. Ruggera are with the Division of Electronic Products, Bureau of Radiological Health, Rockville, Maryland.

minute or less. After the lead is detected, the acid can be washed away before any damage is done to the specimen. The reaction on the glass specimen produces a colored compound distinctive of lead even in the presence of small amounts of other metals such as barium, calcium, copper, manganese, iron, nickel or chromium, some of which might have been added to the molten glass in order to introduce color. Glass samples tested showed 0-50 percent lead.

Preparation of reagent

The dilution is performed by mixing one volume of 48 percent hydrofluoric acid with three volumes of distilled water to make 12 percent hydrofluoric acid. Add 0.005 grams of sodium iodide to each ml of diluted hydrofluoric acid to prepare the final reagent. The reagent may be kept indefinitely in a stoppered polyethylene bottle in the dark. The faint yellow color that might form due to liberated iodine will not interfere with the test. The specimen to be tested should be perfectly clean and dry; the surface should be horizontal and the testing carried out on a bench or table adjacent to a sink with running water. Always keep the reagent in polyethylene containers as hydrofluoric acid attacks glass. Bottles containing the reagent should be opened for only a minimum time necessary to carry out the test. Care should also be taken not to expose the skin to splashes of the acid or its vapor. Any acid inadvertently spilled must be washed away with a large volume of water at once.

Procedure

Apply one drop of the reagent on a convenient spot of the specimen with a polyethylene dropper and observe any color changes or precipitation. On curved surfaces, a small circle of silicone grease will enclose the drop and minimize spreading. If a considerable amount of lead is present, there will be an immediate precipitate of yellow lead iodide in the test drop. If only a trace is present, 2 percent or less, the drop will remain clear for a second or two and then a buff to yellow turbidity will form. A confirmation test that the yellow precipitate is lead iodide can be obtained by washing the yellow precipitate into a polyethylene beaker and adding to the washings a few crystals of NaI or KI. The yellow precipitate of lead will dissolve giving a colorless liquid. If the specimen is a lime glass and has no lead, a white precipitate of calcium fluoride will appear on the surface.

An approximate estimate of the lead content was made by comparing the intensity of the yellow precipitate with standards. The speed of development and the depth of color are a guide to the amount of lead present. The standards were prepared from powdered samples of glass containing known amounts of lead. The powder was spread over the surface of a circular alumina disc having an area of 1 square cm and a thickness of 1 mm (Coors Porcelain Company, Golden, Colorado). The disc was prepared by coating

it with a thin film of a 5 percent Aerosol OT solution (75 percent Acqueous) and then spreading 80 mg of the glass powder over the wetting agent. Percentages of the various glass powders ranged from 0-50 percent PbO. The alumina discs were placed in a cold muffle furnace and the temperature raised to 800° C. This temperature was maintained for 10 minutes and then the discs were allowed to cool. A smooth glaze over the entire surface of the alumina disc resulted. Each alumina button was treated with one drop of the reagent, placed in an oven at 105° C for 10 minutes and then cooled to room temperature.

The reagent produces a slight hazing of the surface of lime glass specimens and a spot on the specimen that is not optically important should be used for testing. Different glasses show different resistance to attack by the reagent depending on their composition. As the PbO content of the glass is increased, the SiO_2 and CaO content are decreased, while the Na_2O and Al_2O_3 content generally remain constant.

Discussion

Table 1 presents a comparison of the estimated amount of lead using the reagent drop method and the amount of lead determined by conventional methods in the various glass specimens analyzed. The lead content of the glass envelope in the various shunt regulator tubes was determined by atomic absorption spectrophotometry (5).

The chemical composition of the various glass specimens is listed in table 2. The major constituents of the glass specimens were determined gravimetrically (6) and by atomic absorption spectrophotometry (5). These data show the relationship between the lead content and other major constituents, such as silica. The silica content of the glass sample is decreased considerably when the lead content is increased. Several shunt regulator tubes, both of the earlier and the more recent version, were investigated to evaluate the usefulness of the lead determination described in this manuscript, in determining a relative correspondence between the amount of lead present in a glass envelope and the resultant X-ray emission from the tube. The tubes were used to expose four 14 in. x 17 in. film packs made up of a KK type and a commercial type film (Eastman Kodak Type S0142, Type S0125, Duo-Ready Pack, Rochester, New York). The film pack was formed into a 17 in. high cylinder. When placed around the tube, the distance from the tube center line to the film was approximately 5 cm. Exposure was made at 30,000 volts and 0.5 milliamperes for a period of six hours. The film was then developed under controlled conditions (new chemicals, constant temperature, gas burst agitation) so that exposure rate could be determined using a densitometer and a set of calibration curves which relate density to exposure rate. The calibration of the film pack was performed by a free air ion chamber.

TABLE 1

A Comparison of Lead Content in Glass Specimens Determined by Conventional Methods and by the Reagent Drop Method

Sample Identification	Determination by Conventional Methods Percent PbO	Approximate Estimation by Reagent Drop Method Percent PbO
Corning Glass Tube Envelope (E-1)	26.47(1)	25
Corning Glass Tube Envelope (E-2)	Trace(1)	0
#3720 TV Picture Tube Safety Panel (SP-1)	0(1)	0
#3755 or 3770 TV Picture Tube Safety Panel (SP-2)	4.87(1)	5
Corning 9019 TV Picture Tube Face Plate	Trace(1)	0
G.E. 6EH4 (GEH-020)	30.32(2)	30
G.E. 6EL4 (OWE-006)	30.52(2)	30
Sylvania 6BK4C (EMP-006)	50.06(2)	50
G.E. 6EL4 (OWE-117) New Type	50.35(2)	50

(1) Determined gravimetrically.
(2) Determined by atomic absorption spectrophotometry.

TABLE 2

Chemical Composition of Glass Specimens
Used in Color TV Receiver Components

Sample Identification	Percent by Weight								
	PbO	SiO_2	CaO	MgO	BaO	Al_2O_3	Fe_2O_3	K_2O	Na_2O
Corning glass tube (E-1)	26.47	55.41	0.26	0.19	0.0	1.65	0.22	$K_2O + Na_2O = 11.72$	
Corning glass tube (E-2)	Trace	72.96	5.01	3.39	0.0	1.93	0.42	$K_2O + Na_2O = 16.45$	
#3720 TV picture tube safety panel (SP-1)	0	68.90	7.32	2.75	0.0	3.08	0.10	$K_2O + Na_2O = 16.82$	
#3755 TV picture tube safety panel (SP-2)	4.87	70.69	6.42	3.60	0.0	$Al_2O_3+Fe_2O_3=1.67$		$K_2O + Na_2O = 12.43$	
Corning 9019 TV picture tube face plate	Trace	65.15	4.75	1.41	6.56	$Al_2O_3+Fe_2O_3=5.49$		$K_2O + Na_2O = 16.49$	
G.E. 6EH4 (GEH-020)	30.32	55.21	<.01	<.01	0.0	2.46	0.45	10.28	3.68
G.E. 6EL4 (GWE-006)	30.52	56.42	0.0	0.0	0.0	1.36	0.0	9.38	3.16
Sylvania 6BK4C (EMP-006)	50.06	37.28	1.22	0.04	0.0	3.36	0.01	8.62	1.28
G.E. 6EL4 (GWE-117) New Type	50.35	37.31	2.14	1.06	0.0	1.33	0.08	7.42	0.74

Table 3 presents a comparison between the amount of lead present in the glass envelope and its influence on exposure rate. Increasing the lead content between the two thinner glass envelopes of tubes 6EH4 and 6BK4C reduced the exposure rate by a factor of 40. The other variable, the wall thickness of the envelope, also had some effect. Comparing the 6EH4 with the 6EL4, a reduction factor of 3.5 is observed. Cross comparisons with the new 6EL4 were not made since a density of 0.01 is not a point on the calibration curve, and the exposure rate was estimated by extrapolating the curve to that value.

In summary, the field test for lead content of the shunt regulator tube could be a valuable tool in field surveys of sets found to emit excessive radiation. When a field of radiation is detected in the area of the shunt regulator tube, the investigator cannot be sure of the exact source since it could be the shunt, the high voltage rectifier, or the picture tube. Using the test kit, he can determine with relative certainty, the lead content of the shunt regulator tube. If the tube contained approximately 50 percent PbO, then it could be eliminated as the defective component. This leaves the high voltage rectifier, which is always in a metal cage for voltage protection and the picture tube. Since instrumentation is available by which one can determine the beam area, the elimination of the shunt regulator from consideration will almost certainly pinpoint the defective component. The picture tube will most probably have a large area beam, whereas the high voltage rectifier would emit a beam only through the air holes in the cage and, therefore, be much smaller in area. A similar set of criteria can be set up for components other than the shunt regulator tube to determine the relative comparison of X-radiation to lead content.

TABLE 3

Relative Comparison Between Lead Content
of Glass Envelope and Resultant Exposure Rate

Tube Type	Average (in.) Envelope Thickness	Chemical Analysis of % PbO	Fast Method % PbO	Net Density of KK Film from Anode Beam	Dose Rate (mR/hr)
G.E. 6EH4 GEH-020	0.035	30.32	30	3.78	11.7
G.E. 6EL4 OWE-006	0.050	30.52	30	1.10	3.33
Sylvania 6BK4C EMP-006	0.034	50.06	50	0.12	0.30
G.E. 6EL4 OWE-117 New Type	0.047	50.35	50	0.01	0.04[a]

[a]This value was determined by extrapolation of the calibration curve.

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compared with standards prepared from powdered samples of glass containing known amounts of lead. The powdered glass was spread over the surface of a circular alumina disc having an area of 1 square centimeter and a thickness of 1 millimeter. Percentages ranged from 0-50 percent PbO. The alumina discs were heated in a muffle furnace at 800° C for 10 minutes and allowed to cool resulting in a smooth glazed surface. A comparison showing the estimated amount of lead using the reagent drop method and the amount of lead determined by conventional methods in the various glass specimens is presented. Several shunt regulator tubes from different manufacturers were investigated to determine a relative correspondence between the amount of lead present in the glass envelope and the resultant X-ray emission from the tube.

KEY WORDS: Lead, Glass, Television, Tubes, Rectifier, Fluoroscopic, Exposure, Methodology, Reagent, Rapid, Screens

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